



Aalborg Universitet

AALBORG UNIVERSITY  
DENMARK

## **Optimisation of biogas production at industrial scale through application of Process Analytical Chemometrics**

*full-scale implementation of process sampling and multivariate monitoring*

Madsen, Michael; Holm-Nielsen, Jens Bo; Esbensen, Kim

*Publication date:*  
2009

*Document Version*  
Publisher's PDF, also known as Version of record

[Link to publication from Aalborg University](#)

*Citation for published version (APA):*

Madsen, M., Holm-Nielsen, J. B., & Esbensen, K. (2009). *Optimisation of biogas production at industrial scale through application of Process Analytical Chemometrics: full-scale implementation of process sampling and multivariate monitoring*. Poster presented at Biogasseminar 2009 (Energinet.dk), Fredericia, Denmark.

### **General rights**

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal -

### **Take down policy**

If you believe that this document breaches copyright please contact us at [vbn@aub.aau.dk](mailto:vbn@aub.aau.dk) providing details, and we will remove access to the work immediately and investigate your claim.

# Optimisation of biogas production at industrial scale through application of Process Analytical Chemometrics

— full-scale implementation of process sampling and multivariate monitoring

**Michael Madsen\*, Jens Bo Holm-Nielsen, Kim H. Esbensen**

ACABS Research Group, Esbjerg Institute of Technology, Aalborg University

Niels Bohrs Vej 8, DK-6700 Esbjerg, Denmark



Web: [www.acabs.dk](http://www.acabs.dk), [www.aau.dk](http://www.aau.dk), [www.aau.dk](http://www.aau.dk)

E-mail\*: [mima@bio.aau.dk](mailto:mima@bio.aau.dk)



## Introduction and aim of the studies

Anaerobic digestion, commonly known as biogas formation, is a complex biological process involving the simultaneous action of multiple groups of microorganisms aiming at acquiring chemical energy for maintaining growth. The mixed bacterial culture can extract energy from a wide variety of organic substrates by transforming carbon into its most oxidised form (carbon dioxide) and most reduced form (methane); the latter being a renewable energy carrier. [1] Process conditions such as temperature, pH, organic loading rate etc. have to be carefully controlled in order to maintain a stable biogas production. Hence, it is desirable for the biogas plant operator to be able to monitor the current state of the process, since it allows him to launch proper countermeasures, for instance diluting the feed, if a critical trend becomes apparent from the measured process variables. [2]

Numerous scientific laboratory investigations conducted over the past decades have indicated that the key to optimise the biogas process lies in monitoring, and controlling, a few chemical parameters. [2]

These chemical constituents are intermediates stemming from the core conversion mechanisms of the biogas process depicted in figure 1.

Investigations carried out at Aalborg University, Esbjerg Institute of Technology have proven that multivariate methods such as near infrared spectroscopy and passive acoustic monitoring combined with powerful mathematical and statistical methods (chemometrics) successfully can decipher the vast amount of chemical information in the complex bioslurry and reveal the state of the process with respect to the most important parameters (content of dry matter, organic dry matter, ammonium, and short chain volatile fatty acids). [3-4]

This Ph.D. research project seeks to bring these findings from the laboratory to the market through a full-scale implementation in the industry by applying the scientific competences of ACABS Research Group: representative sampling and process sampling, applied chemometrics, and applied biotechnology and Green Engineering.

## The biogas formation process

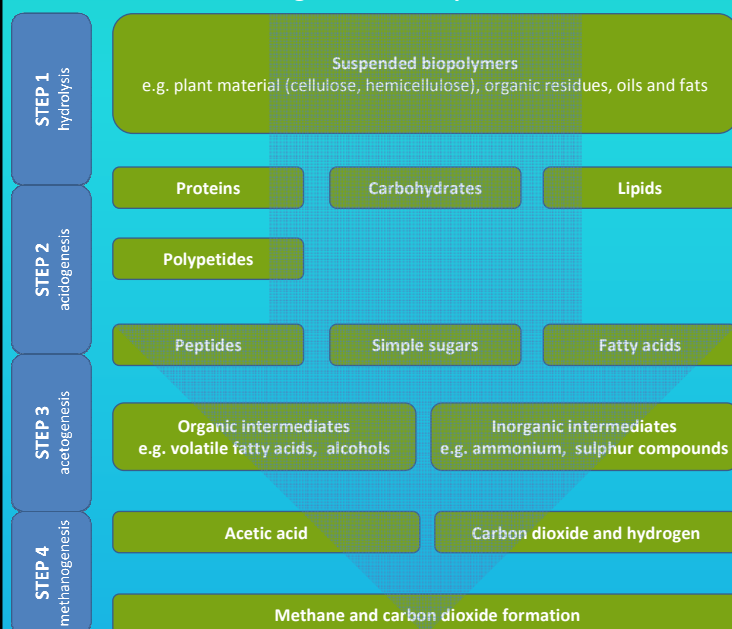
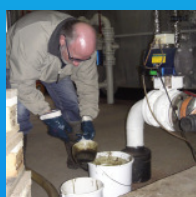


Figure 1. Schematic representation of the biogas formation process  
Adapted in modified form from [1]

## Process monitoring by Process Analytical Chemometrics vs. a centralised laboratory strategy

extraction of physical sample and  
representative mass reduction if needed

sample analysis by  
multivariate sensors in or at  
the process stream



conventional sample  
analysis at a  
centralised laboratory



decision on process adjustment  
based on analytical result



seconds to  
minutes later...

hours to  
days later...

Figure 2. Process Analytical Chemometrics vs. a centralised laboratory strategy for process monitoring

## Principles of Process Analytical Chemometrics

As shown in figure 2 on the left, there is a tremendous difference between asserting the state of a process by using a conventional centralised laboratory strategy and by implementing Process Analytical Chemometrics (PAC). Where conventional centralised laboratory analysis typically is used to verify that the quality of an end product is within specifications, PAC enables the plant operator to adjust the production process continuously. Hence, PAC generates an enormous amount of real-time process data that can be subjected to comprehensive data analysis enabling an in-depth understanding of the process at hand and its dynamic nature. This is something that the conventional centralised approach never will be able to do. At the same time PAC reduces costs for chemical reference analysis substantially, since the amount of physical samples that need to be analysed are reduced to periodical validation samples.

PAC combines the power of three fundamental disciplines:

- Δ representative sampling
- Δ advanced sensor technology
- Δ multivariate data analysis

Please consult the literature for details on these three subjects and their synergies. [5-6]

## Literature references

- [1] Mata Álvarez, Macé, Llabrés. *Anaerobic digestion of organic solid wastes. An overview of research achievements and perspectives*, *Bioreour. Technol.* (2002), pp. 3-16, doi: 10.1016/S0960-8524(00)00023-7
- [2] Pind, Angelidaki, Ahring, Stamatiadou, Lyberatos, *Monitoring and control of anaerobic reactors*, *Advances in Biochemical Engineering/Biotechnology* (2003), pp. 135-182, doi: 10.1007/3-540-45838-7
- [3] Holm-Nielsen, Lomborg, Oleskowicz-Popiel, Esbensen. *On-line near infrared monitoring of glycerol-boasted anaerobic digestion processes: evaluation of Process Analytical Technologies*, *Biotechnol. Bioeng.* (2008), pp. 302-313, doi: 10.1002/bit.21571
- [4] Lomborg, Holm-Nielsen, Oleskowicz-Popiel, Esbensen. *Near infrared and acoustic chemometrics monitoring of volatile fatty acids and dry matter during co-digestion of manure and maize silage*, *Bioreour. Technol.* (2009), pp. 1711-1719, doi: 10.1016/j.biortech.2008.09.043
- [5] Esbensen and Paasch-Mortensen, *Process Sampling: Theory of Sampling – the missing link in Process Analytical Technology (PAT)*, Chapter 3 in: Bakeev, K. (Ed.), *Process Analytical Technology*, 2<sup>nd</sup> ed., 2009, Blackwell Publ.
- [6] Esbensen and Julius, *Representative Sampling, Data quality, Validation – A necessary Trinity in Chemometrics*, In: Brown, S., Tauler, R., Walczak, R. (Eds.), *Comprehensive Chemometrics*, vol. 4, 2009, pp. 1.20, Oxford, Elsevier

## Planned project deliverables

- Review of existing, commercial technologies for online process monitoring
- Review and critical evaluation of existing sampling equipment and procedures
- Demonstration of multivariate methods in full-scale (spectroscopy and acoustic)
- Implementation of representative sampling mechanism in full-scale
- Implementation and validation of multivariate monitoring in full-scale

## Acknowledgements